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Fabrication and Characterisation of Recycled Polyethylene Terephthalate/Graphene Oxide Nanofibres as a Potential Adsorbent for Methylene Blue

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Polymeric nanofibres can be good dye adsorbents as they have large surface areas, good connectivity, and tunable wettability. In this study, recycled polyethylene terephthalate (rPET) nanofibres loaded with graphene oxide (GO) in various amounts (0 - 1.5 v/v%) were fabricated using the electrospinning technique. GO is a carbonaceous material that exhibits a large specific area and contains an oxygenous functional group that significantly enhances the performance of electrospun nanofibres. Scanning electron microscopy images showed that rPET/GO had smooth fibres with diameters as low as 118 ± 56 nm. Fourier-Transform Infrared Spectroscopy (FTIR) confirmed GO had been well incorporated in the rPET nanofibres. The use of GO as a filler increased the tensile strength to 32.84 ± 0.33 MPa, and thus enhanced the mechanical properties of the polymeric nanofibres. The adsorption capacity of the rPET/GO nanofibres for methylene blue (MB) was evaluated, and rPET nanofibres loaded with GO showed better adsorption compared with pure rPET nanofibres. From this study, rPET/GO nanofibres show good potential as adsorbents for the treatment of dyes in wastewater.

Key words: Nanofibers; electrospinning; graphene oxide; adsorption; methylene blue

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The textile, paint, and plastic industries have negatively influenced the quality of freshwater, the environment, and human health by emitting hazardous effluents such as synthetic dyes [1-3]. A synthetic dye such as methylene blue (MB) poses a significant threat to aquatic life due to its deep colour and carcinogenic effect. It also reduces the level of dissolved oxygen (DO), resulting in higher biochemical oxygen demand (BOD) [4-5]. Therefore, the removal of MB is of great importance. Several methods to remove the synthetic dye from wastewater include adsorption, membrane filtration, ion exchange, and coagulation [6]. Among these methods, adsorption is the most effective due to its efficiency, simple design, ease of use, low cost, and varied adsorbent materials [6,7]. Based on previous studies, cheap and natural adsorbents such as clay, rice husk, and orange peel have been used, but the results were not satisfactory [8]. Recently, a new synthetic polymer adsorbent made from nanofibres has been extensively studied. These nanofibres can be fabricated using the electrospinning technique and offer unique properties such as a significant surface area per mass ratio, high porosity, and high permeability. The controllable properties of these nanofibres allow for their good performance in wastewater treatment [9-11].

Polyethylene terephthalate (PET) solid waste, which is available in abundance, can be recycled into electrospun nanofibres for use in wastewater treatment, clothing, and biomedical applications [12-14]. Zander et al. [15] successfully prepared rPET electrospun nanofibres for microfiltration applications, e.g., as a prefilter in a wastewater treatment system. rPET nanofibre filters have shown great promise as a replacement material for commercial membranes. However, rPET has issues such as degradation and hydrophobicity that limit its adsorption capacity [16]. Thus, incorporating graphene-based nanomaterials into the polymer matrix maintains the nanofibres' permeability and enhances its surface hydrophilicity for better adsorption [17-20]. Graphene oxide (GO) is a carbonaceous material derived from a monolayer of carbon atoms organized in a hexagonal graphene ring. The presence of oxygen-containing groups such as hydroxyl, carboxyl, carbonyl, and epoxide on the graphene surface indicates the successful synthesis of graphene oxide. A single-layer atom structure of

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graphene oxide potentially provides a large surface area for methylene blue adsorption [21-24]. Swaminathan et al. [23] conducted a study on the effectiveness of polyacrylonitrile (PAN) yarn waste/GO nanofibres in removing methylene blue. They found that the GO-loaded nanofibres were able to adsorb methylene blue significantly better than PAN yarn waste alone. According to Chia et al. [25], GO can adsorb cationic dyes from non-acidic wastewater to a maximum capacity of 700 mg/g. In this study, we fabricated electrospun rPET nanofibres incorporated with GO to investigate its potential for use as MB adsorbents. From what we know, the combination of rPET/GO nanofibres has not yet been studied as potential MB adsorbents. The morphology, mechanical properties and MB adsorption capacity of the rPET/GO nanofibres were examined. The use of electrospun polymeric nanofibres created from sustainable recycled materials is a good way to turn waste into higher-value products in the field of environmental analysis.

MATERIALS AND METHODS

1. Materials

A recycled SPRITZER® PET bottle was used as received. Graphene oxide was obtained from the Advanced Material Lab, Kulliyyah of Science, IIUM Kuantan. Trifluoroacetic acid (TFA) and dichloromethane (DCM) were purchased from Sigma-Aldrich, Germany, while ethanol, 70% was purchased from HmbG Chemicals, Germany. Methylene blue was purchased from Fisher Scientific, United Kingdom. All chemicals used in the study were of analytical grade and used without further purification.

2. Fabrication of rPET/GO Nanofibres

15 wt% rPET samples were dissolved in 1:1 v/v TFA/DCM and stirred at 150 rpm under room temperature for 72 hours until completely homogenous. 5 wt% GO was dissolved in 1:1 v/v TFA/DCM and sonicated for 10 minutes to obtain a good dispersion. Approx. 0.5 to 1.5 % (v/v) of GO stock solution was added dropwise into the respective rPET solutions while stirring. The rPET loaded with GO was sonicated for 10 minutes to ensure the GO homogenously dispersed in the rPET solution. The samples were continuously stirred until further use. The rPET/GO solution was transferred to a 3 mL syringe and attached to a stainless-steel needle with an inner diameter of 23G. The electrospinning parameters used were 0.7 mL/h flow rate, 15 kV, and 15 cm as a tip to distance. The samples were fabricated using the electrospinning technique at room temperature (27± 5°C) and 65% humidity. The nanofibres were collected on aluminium foil and dried overnight in a desiccator to prevent contamination from the surrounding environment.

3. Characterisation of rPET/GO Nanofibres

The morphology of the electrospun rPET and rPET/GO nanofibres was examined by SEM (Carl Zeiss/Evo 50). A small section of the nanofibre sample was mounted on a brass stub using double-sided tape and then sputtered with gold using a sputter coater (Leica EM SCD005). All samples were analysed under 5000x magnification. Around 100 rPET and rPET/GO nanofibres at five different spots were measured using Image J software (Java 1.8.0_172). An FTIR spectrophotometer (PerkinElmer) equipped with an overhead ATR accessory was used for analysing the chemical structure of the rPET and rPET/GO nanofibres within the range of 4000 to 400 cm^{-1}. The FTIR spectra were plotted using OriginPro software. Tensile testing was conducted to determine tensile stress and elongation at the break. The testing was performed by using 20 N load cells (Model UUK 5, Korea) equipped with a micro-stepper motor system (Ezi Step, Fastec, Korea) and OMRON RX-RX25 data logger to file and record the loads. Before testing, the rPET and rPET/GO were cut into rectangular shapes (5×15mm) with 10 mm gauge length. The elongation was determined by a 1.0 mW Omron laser detector with a detection limit of 2.5 ms^{-1}/-1-1600nm. The tensile test was carried out at 1 mm/min crosshead velocity. The average maximum load and graph of every sample was calculated using Sigmaplot Software (ver.10.0). Samples were measured in triplicate to obtain average values for the tensile test.

A 1000 ppm stock standard solution of MB dye was prepared and used for the adsorption capacity experiments. Further dilutions were carried out for various concentrations of MB. The concentrations of the MB solution pre- and post-adsorption were measured using a UV-vis spectrophotometer (Lambda 35, PerkinElmer, USA) at a maximum wavelength of 660 nm. Batch adsorption experiments were carried out using rPET, rPET-GO (0.5), rPET-GO (1.0), and rPET-GO (1.5) nanofibres with different weights (10, 20, 30 mg) in 20 mL of 10 ppm MB solution. The final MB concentration was determined through calibration with different initial concentrations of MB. Each experiment was repeated in triplicate, and the results were given as averages. The adsorption efficiencies and intake capacities were calculated using equations (1) and (2) as follows:

$$Adsorption\ Efficiency\ (\%) = \frac{C_i - C_f}{C_i} \times 100 \quad (1)$$

$$q_e = \frac{(C_i - C_f)V}{W} \quad (2)$$

where C_i (mg/L) is the initial concentration of MB solution, C_f is the final concentration of MB solution, v (L) is the volume of MB solution used, and w (g) is the weight of nanofibres.

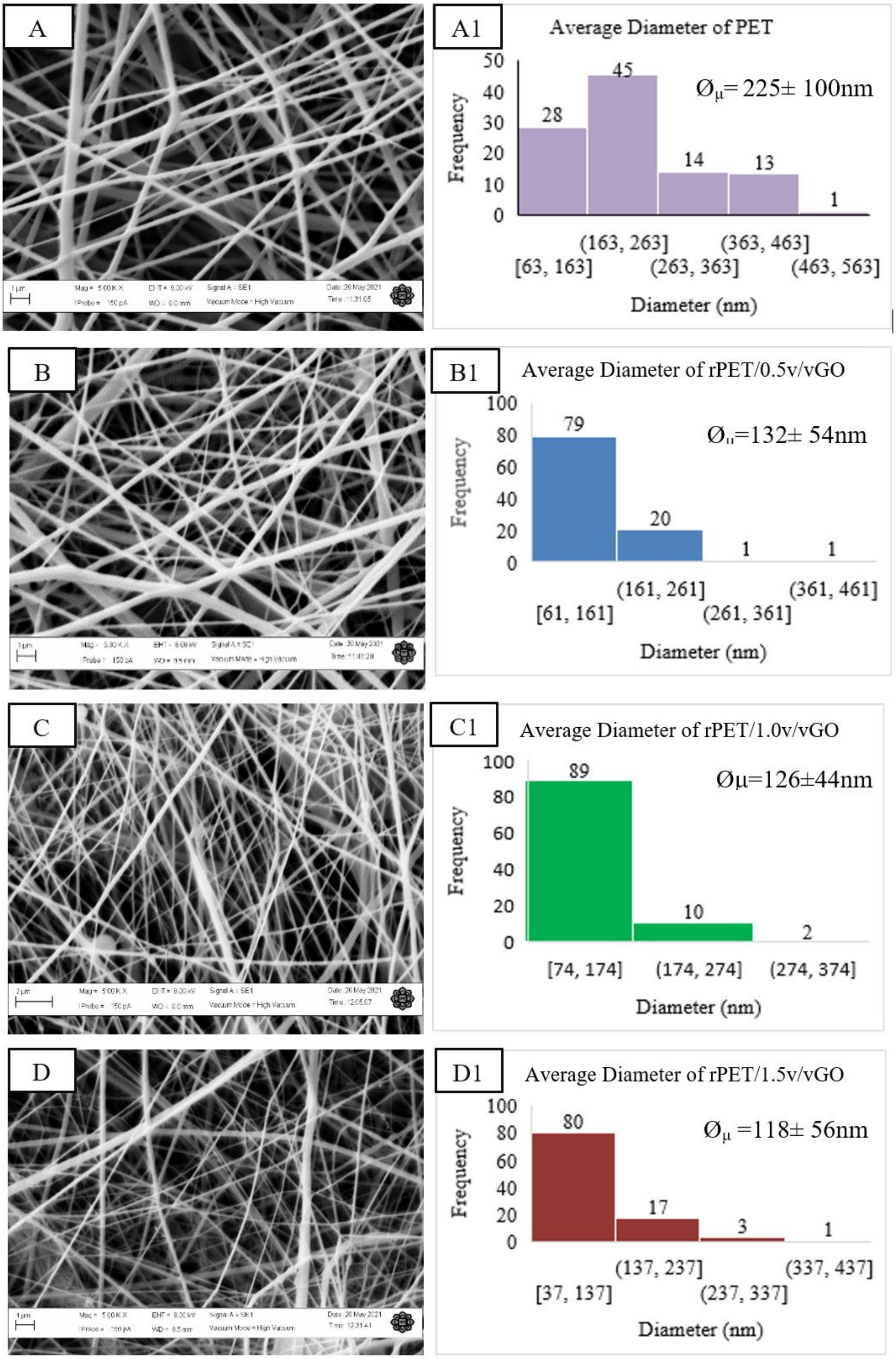

Figure 1. SEM images and histograms of 15 wt% rPET at various GO concentration: A) rPET; B) rPET/0.5 GO; C) rPET/1.0 GO; D) rPET/1.5 GO at 5000x magnification [Fixed parameters:15 kV, TCD:15 cm, flow rate: 0.7 mL/h].

RESULTS AND DISCUSSION

1. Morphology of rPET/GO Nanofibres

Figure 1 represents SEM images of rPET and rPET loaded with 0.5, 1.0 and 1.5 GO nanofibres. The rPET nanofibres (Figure 1(A)) were abundant, continuous and uniform in size with an average diameter of 225 ± 100 nm. rPET loaded with 0.5 % (v/v) GO (Figure 1 (B)) showed smooth and even nanofibres with an average diameter of 132 ± 54 nm. rPET with 1.0 % v/vGO (as in Figure 1 (C)) showed thinner nanofibres of irregular size with an average diameter of 126 ± 44 nm. When GO was increased up to 1.5 % (v/v), the nanofibre sizes were more uneven (Figure 1 (D)), with an average diameter of 118 ± 56 nm. Loading GO up to 1.5 % v/v in the rPET nanofibres decreased the fibre diameter due to increased electrical conductivity in the polymer solution. Similar trends have been reported by other researchers [26-28]. When the electrical conductivity is high and with a fixed high voltage, the electrospinning jet undergoes more stretching and elongation, leading to smaller and more uneven fibre diameters upon addition of GO [26,29, 30].

2. Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR spectra of GO, rPET and rPET nanofibres loaded with various concentrations of GO are shown in Figure 2. The characteristic peaks for rPET and GO are listed in Table 1. GO appeared as broad peaks at 3420 cm^{-1} due to O-H stretching, at 1730 (C-O) and 1615 cm^{-1} indicating aromatic C=C groups, and 1080 cm^{-1} for C-O-C groups.The main peaks of rPET appeared at 2930 cm^{-1} (C–H stretching), 1720 cm^{-1}(C=O carboxylic acid group stretching),1615 cm^{-1} and 1505 cm^{-1} (aromatic C=C), 1420 cm^{-1} (C-O stretching), 1240 cm^{-1} (C–C–H aromatic ring stretching), 1174 cm^{-1} (C–O tertiary alcohol stretching). When rPET was loaded with various amounts of GO, a broad peak appeared around 3600 - 3420 cm^{-1}, corresponding to O-H stretching, which indicates the existence of hydrogen bonding between the carboxyl group of rPET and the hydroxyl group of GO [27]. Hence, it proves the successful deposition of GO on the rPET nanofibres.

3. Tensile Test

The mechanical properties of the rPET/GO nanofibres determine their ability to bear a load while adsorbing MB. The effect of GO on the mechanical properties of rPET nanofibres was evaluated based on its tensile strength. Figure 3 shows that the tensile strength of pure rPET nanofibres was 11.13 ± 1.3 MPa. When 0.5 and 1.0 % v/v/ GO were added into rPET, the tensile strength increased to 27.96 ± 0.85 MPa and 32.84±0.33 MPa, respectively. Incorporating GO up to 1.0 v/v in rPET nanofibres increased the tensile strength due to the homogenous dispersion of GO within the rPET solution [26, 31]. GO was expected to improve the mechanical strength but at a certain threshold amount, the mechanical properties began to deteriorate. The addition of 1.5 v/v of GO suddenly decreased the tensile strength of rPET nanofibres to 23.58 ± 0.23 MPa; this was due to the partial agglomeration and poor dispersion of GO in the rPET nanofibres [26, 28].

Table 1. Characteristic peaks of rPET and GO [26,27]

rPET (cm^{-1})	GO (cm^{-1})	Bond vibration
-	3420	O-H stretching
2930	-	C–H stretching
1720	1730	C=O carboxylic acid stretching
1615, 1505	1615	C=C aromatic
1420	-	C-O stretching
1240	-	C–C–H aromatic ring stretching
1174	-	C–O stretching tertiary alcohol
-	1080	C-O-C stretching

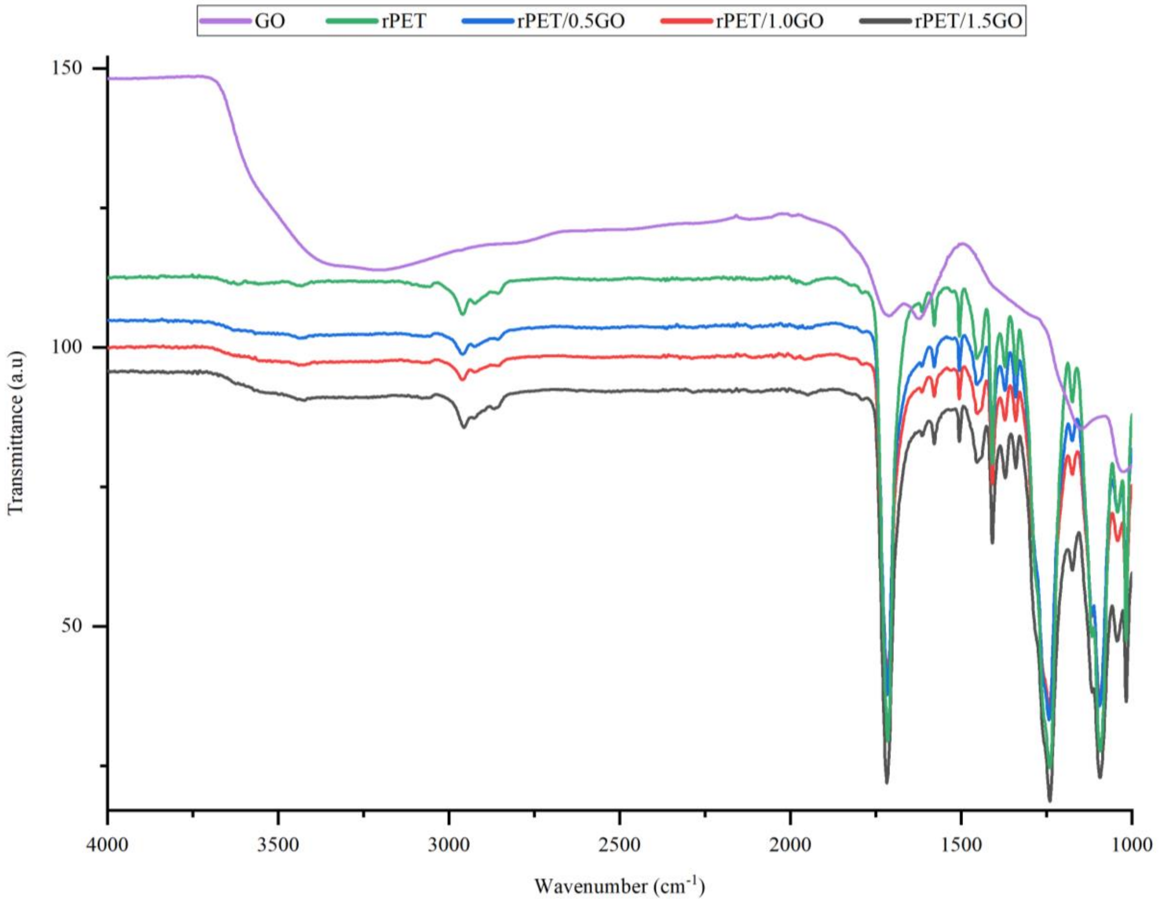

Figure 2. FTIR spectra of GO, rPET, rPET/0.5GO, rPET/1.0 GO, and rPET/1.5 GO nanofibres.

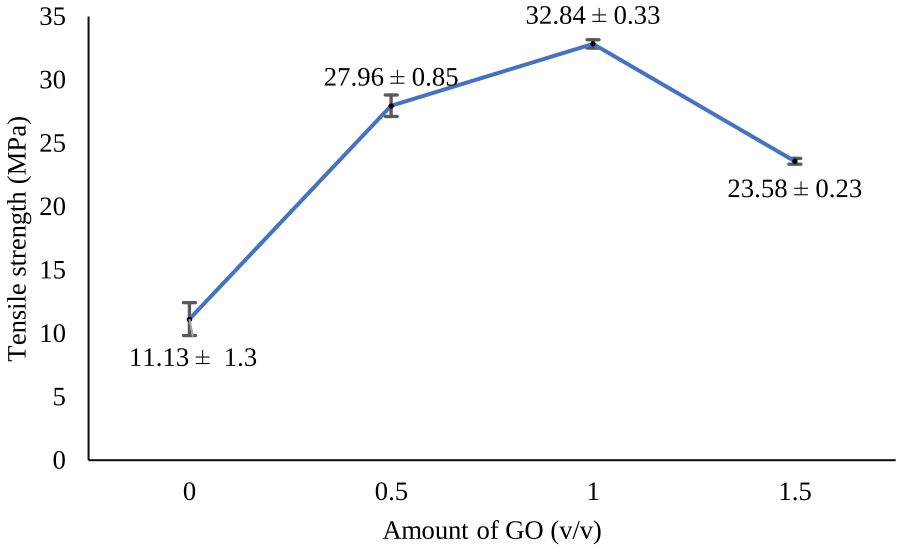

Figure 3. The tensile strength of rPET, rPET/0.5v/v GO, rPET/1.0v/v GO and rPET/1.5v/v GO

4. Adsorption Studies

The effect of mass on adsorbent efficiency was measured and presented in Figure 4 below. According to the Beer-Lambert law, absorbance is directly proportional to the concentration of the absorbing analytes at a constant path length [32]. So, the higher the absorbance of MB, the more concentrated the methylene blue solution. Simultaneously, the adsorption of MB by the respective nanofibres decreased [23].

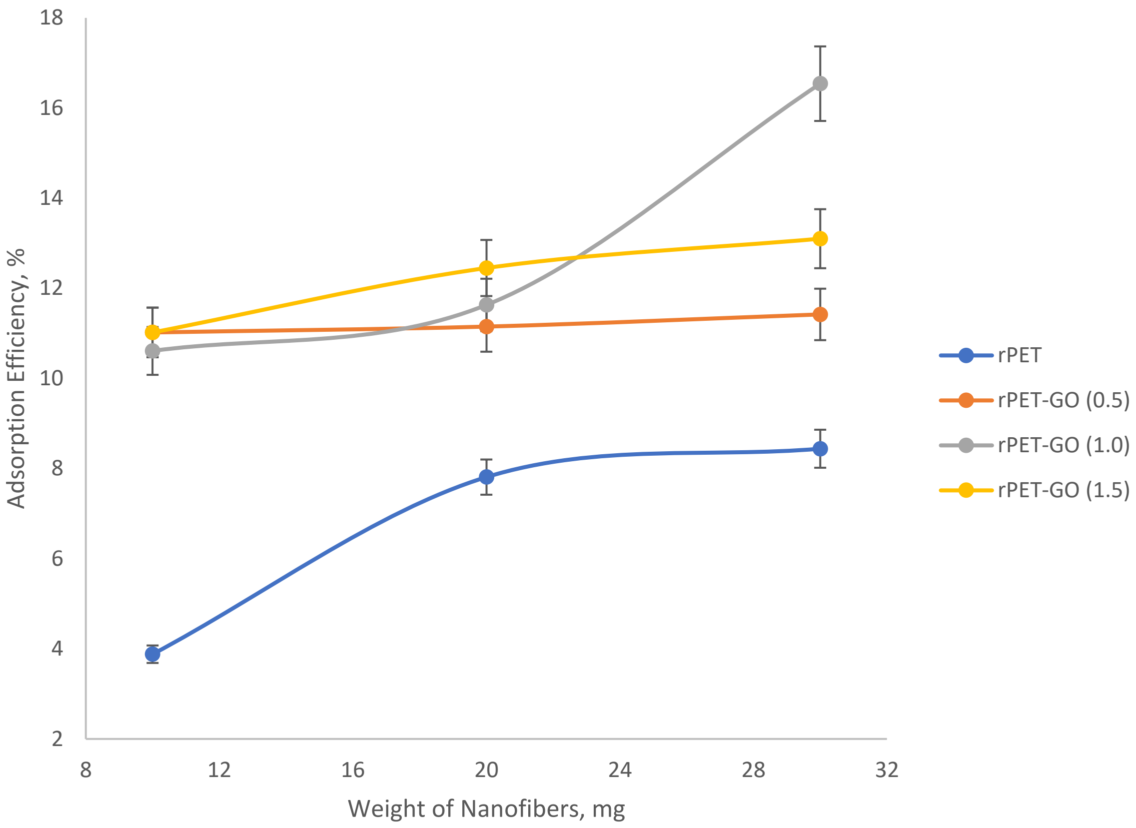

Figure 4. Adsorption Efficiency of rPET and rPET-GO nanofibres

Figure 4 shows that the rPET/GO nanofibres had higher adsorption capacities than pristine rPET nanofibres at all mass ranges. It indicates that the addition of GO improved the efficiency of the adsorbent. PET is hydrophobic because it lacks hydrophilic functional groups [33]. In contrast, GO is highly hydrophilic and contains a high density of hydroxyl, carboxyl, carbonyl, and epoxide groups per surface area, leading to a higher negative charge density on its surface [23]. Based on Figure 2, GO was incorporated well in the rPET nanofibres and slightly reduced the hydrophobic properties of rPET. The oxygenated groups interacted with MB via hydrogen bonds, π–π interactions, and electrostatic interactions and improved the nanofibres' adsorption capacity to adsorb methylene blue (13,17,18). Therefore, as the percentage of GO in the rPET nanofibres increased, the availability of adsorption sites for methylene blue increased simultaneously. As a result, the methylene blue removal efficiency increased [34]. With 10 mg of adsorbent, rPET loaded with various amounts of GO showed almost the same adsorption efficiency, around 10 to 11 %. With 20 mg of adsorbent, the adsorption efficiencies of rPET/1.0 GO, and rPET/1.5 GO were 11.63% and 12.45%, respectively, showing an increasing trend compared to rPET/0.5 GO at 11.15%. With 30 mg of adsorbent, rPET/1.0 GO showed a higher adsorption efficiency up to 16.45 % compared to other samples. rPET/1.5 GO had a lower adsorption efficiency than rPET/1.0 GO, possibly due to having reached its maximum capacity of GO. Thus, increasing the mass of nanofibres promoted adsorption efficiency. With increased mass, a greater surface area is available for dye adsorption [35]. As a result, the adsorption rate increased, but the nanofibres' uptake capacity was decreased.

CONCLUSION

An rPET waste bottle was successfully recycled into polymeric nanofibres using the electrospinning technique. The rPET nanofibres were functionalised with various amounts of GO, which decreased the average fibre diameter to 118 ± 56nm. FTIR analysis confirmed the successful blending of GO in the rPET nanofibres and revealed the existence of hydrogen bonding between rPET and GO. Increasing the amount of GO in the rPET nanofibres improved its mechanical properties to a point. However, incorporating 1.5 v/v of GO lowered the tensile strength due to partial agglomeration and poor dispersion of GO in the polymer solution. rPET functionalised with GO could simultaneously minimise the environmental problems caused by hazardous MB and waste rPET bottles. Further optimisation of the adsorbent surface is needed to determine the most efficient adsorption process. The determination of adsorption efficiency with varying parameters such as temperature, pH, and contact time would be useful. The adsorption kinetic model should also be studied to obtain more information on adsorption rate, adsorbent performance, and the interaction between adsorbate and adsorbent.

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The authors declare that they have no conflict of interest.

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